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ASSESSMENT OF $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-d}$ AS A CATHODIC MATERIAL FOR SOLID OXIDE FUEL CELL APPLICATIONS

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ABSTRACT

The features and performance of each device and equipment is directly related to its composing materials. The efficiency and durability of the Solid Oxide Fuel Cells (SOFC) also depend on their composing elements. Other important factors for the SOFC behavior are the working conditions (compositions, and humidity of the fuel and oxidant gaseous mixtures, temperature, pressure, etc.). These facts predetermine the necessity for detailed investigation of the behavior of each composing layer of SOFC. For this reason, the electrical characteristics of $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-d}$ as SOFC cathodic material were evaluated by high temperature electrochemical methods, durability tests, and posterior XRD and SEM characterization. As a result, the optimal composition and working conditions for the investigated material in assembled SOFC were determined. The obtained material reveals excellent durability and compatibility with the rest SOFC components.

Keywords: SOFC, cathodic materials, $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-d}$ thermal stability, durability, Thermal Voltammetry, AFM.

INTRODUCTION

The permanent increment of human population is accompanied by increase of energy demand [1, 2], and more restrictive environmental regulations [3, 4]. In that instance, the Solid Oxide Fuel Cells (SOFC) technology has emerged as an efficient substitution of the presently existing energy devices [5]. In addition to their higher efficiency than those obtained from the traditional energy conversion systems, a fuel cell provides many other advantages like reliability, modularity, fuel flexibility and very low levels of NO_x and SO_x emissions

[6]. In addition, the SOFC can be used for co-generation of electricity and valuable products for the chemical industry [7-10]. Various materials were submitted to investigation as potential SOFC components [11-20].

The present paper is an assessment of the electrical parameters of strontium doped neodymium nickelate at different temperatures, from 700 to 900°C, and evaluation of its behavior as a component of symmetric SOFC.

By definition, the Solid Oxide Fuel Cells could be considered as solid state electrochemical devices for conversion of gaseous fuels into electrical and thermal energy. Fig. 1 represents a schematic view of SOFC with

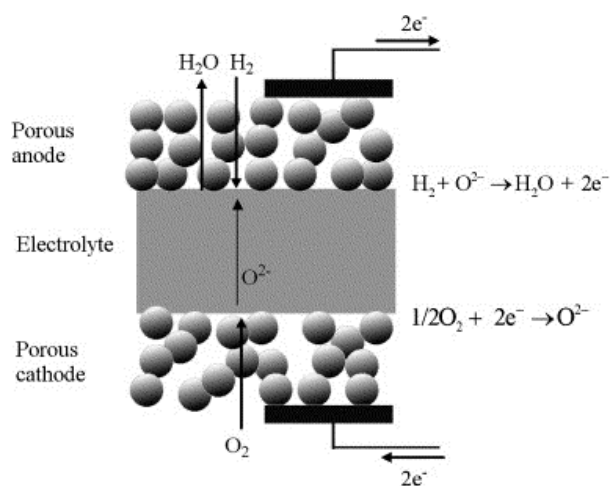


Fig. 1. Schematic presentation of the SOFC process with an O^{2-} transporting electrolyte [1].

an O_2 - transporting electrolyte, according to [1].

Like the classical Galvani elements, described in the literature [21], SOFCs are composed by a cathode (where the oxygen reduction process takes its place), an anode (where oxidation of H_2 or CH_4 is performed), and solid electrolyte (for ionic transport between the electrodes).

EXPERIMENTAL

Cell assembling

$Nd_{2-x}Sr_xNiO_{4-\delta}$ ($x = 0.2$ to 0.8), powders were synthesized using the citrate–nitrate process (CNP) [22]. According to the formula, stoichiometric amounts of $Nd(NO_3)_3$, $Sr(NO_3)_2$ and $Ni(NO_3)_2 \cdot 6H_2O$ analar grade, were mixed in a $H_2O:HNO_3 = 1/3$ to $2/3$ solution. After dissolving, citric acid was added into the nitrate solution at 1,1:1 molar ratio of metal ions/citrates, according to the chemistry of the auto combustion process [21, 24]. After the auto combustion and drying, the resultant cathode powders were calcined in air at 1100, 1150, and 1200°C respectively, for 12 h. Subsequently, the cathode materials were ball milled to particle size about 1,5 μm . All powder samples were controlled by laser granulometry. The powders of gadolinium doped ceria (CGO) and yttrium doped ceria (YDC) were purchased ready-to-use, as well as the yttria stabilized zirconia (YSZ) powder. The phases obtained have been characterized by X-ray diffraction (Rigaku, D/MAX-3B) using $Cu K\alpha$ radiation.

The cathode powders were mixed with terpeneol to form slurry. The same terpeneol slurry was used for

the CGO and YDC interlayer preparation. First the interlayer, and then the $Nd_{2-x}Sr_xNiO_{4-\delta}$ cathode pastes were screen-printed on both sides of the YSZ electrolyte pellet to form an electrode areas of 1.5 cm^2 , as working electrodes (WE) in a symmetrical cell. The cathodes were first slowly heated at 400°C for 2h to eliminate organic binders, followed by sintering at 900°C for 4h in air, with a heating/cooling rate of 3°C min^{-1} .

The electrolyte pellet preparation consists of pressing 1.3 g. of YSZ powder until achieving 90 % of its theoretical density ($\sim 5.7 g/cm^3$). The pressed electrolyte pellets are calcined at 1400°C for 12 h.

The impedance spectra were recorded over the frequency range 1 MHz to 0.1 Hz using Autolab PGStat30, from EcoChemie, Netherlands. The measurements were performed at OCV as a function of temperature (400 – 800°C). The results from the impedance spectra were interpreted and the area specific resistance (ASR) was calculated. After the impedance spectroscopy, the tested cells were observed by scanning electron microscopy (SEM) (Hitachi, S-4700 FEG, Japan), using a standard preparation procedure.

On the anode supported half cells with the Yttria Stabilized Zirconia (YSZ) solid electrolyte a functional cerium galate (CGO) interlayer and cathodic material were deposited via the screen-printing method in I.C.M.C.B. (France).

After each cycle of deposition, every layer was sintered at an individual thermal cycle. The primer CGO layer posterior was treated in the following cycle: increase with 1°C/min up to 400°C, followed by 3°C/min until reaching of 1250°C. The final cooling was performed at 5°C/min. The cathodic layer was treated at 900, 1000, and finally at 1100°C. Thus, the obtained bi-layer film acquired 30 μm of total thickness. After the deposition, the obtained bi-layer film was submitted to the thermal treatment, described hereunder.

Characterization procedures

- Determination of the specific resistance: It was determined by High temperature Impedance Spectroscopy, in the frequency range from 1 MHz to 0.1 Hz, with 100 mV amplitude of the excitation signal, using Autolab PGStat-30. The measurements were performed at Open Circuit Voltage (OCV) in the temperature interval between 400 and 800°C.

- Acquisition of the working characteristics of the obtained cells: It was performed in a high temperature

(furnace), with controlled gas-flows produced by ECN – Netherlands. The experimental procedure was as follows:

1- Initial achieving of the labor temperature in nitrogen atmosphere for reduction of NiO to NiO and stabilizing of the Ni:YSZ – metal-ceramics.

2- Stable charging of the cell up to 0.3 A/cm² and retention of this temperature for 2 hours.

3- Acquisition of the voltammograms: was performed in a Galvani-static regime at 50 mA/cm² for 180 seconds. The voltammograms were obtained at a maximal threshold: 0.420 A/cm² and a minimal threshold: 500mV. The voltammometric measurements were repeated at: 900, 850, 800, 750 and 700°C, respectively.

RESULTS AND DISCUSSION

Determination of the area specific resistance and polarization resistance

After the respective impedance measurements, it was established that the obtained cells with the respective cathodes represent almost the same values of specific resistances for the nine temperatures, in the 400 – 800°C range, as is shown in Fig. 2.

As could be seen (Fig. 2, position (a)) the area surface resistance (ASR) does not change significantly its dependence from the temperature within the 400 – 800°C range. This fact is indication that any sharp

changes (such as cracks or phase transitions) do not appear in this temperature interval. Position (b) depicts the relations of the polarization resistances with the temperature for various Nd_{2-x}Sr_xNiO₄ compounds, where x = 0 up to 0.8. The figure obviously shows that the pure NdNiO₄ reveals the strongest dependence of Rp on the temperature. At 727 °C there is an intersection between the Rp = f(1000 / T) curves of Nd_{1.6}Sr_{0.4}NiO₄ and this of the pure NdNiO₄. Nevertheless, at 496 °C the difference between the Rp values of these substances exceeds an entire order of magnitude. Furthermore, the curves of all Sr-doped compounds possess the same slope. Among them, the Nd_{1.6}Sr_{0.4}NiO₄ is situated below all the rest, indicating that this substance possesses the lowest resistances for the entire temperature interval. As a result, it could be concluded that Nd_{1.6}Sr_{0.4}NiO₄ is the optimal composition among the investigated compounds.

Compatibility of Neodymium nickelate cathodes in assembled cells

The evaluation of the working characteristics was performed on assembled cells with YSZ solid electrolytes and CGO interlayers. The tests were executed at different temperatures in the 700 – 900°C range, with 50°C steps and after different durations of working of the respective cells at 850 °C. In these conditions, the electrical characteristics of the cells were acquired. The results are presented in Fig. 3.

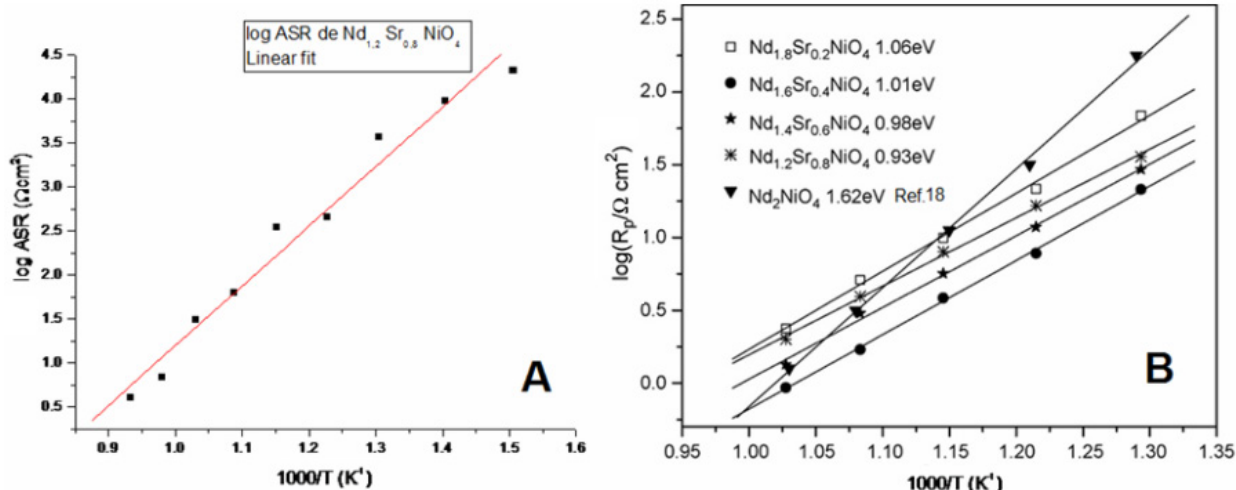


Fig. 2. Temperature dependences of the Area Specific resistance for $\text{Nd}_{1.2}\text{Sr}_{0.8}\text{NiO}_4$ (a); and polarization resistances for $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_4$ (b).

On the basis of the obtained results, it was determined that the highest resistance of $R_p = 0.583 \Omega/\text{cm}^2$ was registered for the cell, submitted to 700°C , whereas the lowest one $R_p = 0.937 \Omega/\text{cm}^2$ belongs to the cell at 900°C . The maximal Open Circuit Voltage (OCV) voltage of 1.08 V was registered for the cell, submitted to 700°C . At elevated temperatures up to 900°C was found a decrement of OCV from 1.08 до 0.99 V . In the temperature interval $700 - 800^\circ\text{C}$, the cell reached the maximal current density of $0.4 \text{ A}/\text{cm}^2$. Although the high values of the voltage, the lower labor temperature results in achievement of a critical value of OCV. It was established that the thermal regime between 850 and 900°C , assists the reaching of maximal current density of $0.4 \text{ A}/\text{cm}^2$. The optimal combination of initial voltage and current density were registered for the cell, submitted to 900°C .

Obviously, the compatibility of the cathodic materials with the rest SOFC components could be assessed by the durability of the entire cells. Fig. 3 (b) shows the change of the electrical parameters after different durations of the work of the SOFC at 850°C . The experiments were performed by charging the cell with $0.5 \text{ A}/\text{cm}^2$, for respectively: 0, 100, 200, 300 and 400 hours. The critical minimum of 0.5 V at maximal current density of $400 \text{ mA}/\text{cm}^2$ was not achieved even after 400 hours of charging. The OCV decreased within the time from 1.04 to 0.999 V , due to increase of losses related to the activation polarization. The most probable reason for these losses is the formation of NiO . This oxide acts as a barrier for the electronic and ionic currents.

Another peculiar feature of the position (b) of the

figure is that the cell reached the lowest current range after 200 h, and afterwards it returned gradually to the initial curve, until reaching an overlapping of the curves for 300 and 400 hours. However, the calculations showed that R_p for the initial period is $0.937 \Omega/\text{cm}^2$, reached $R_p = 1.625 \Omega/\text{cm}^2$ after 100 hours, and stabilized at $R_p = 1.429 \Omega/\text{cm}^2$ after the subsequent 200, 300 and 400 hours of exposition. This fact means that the cell negligibly changes during the initial 100 hours of exposition to 850°C and charging by $0.5 \text{ A}/\text{cm}^2$ and afterwards it retains its characteristics, revealing a tremendous durability.

XRD characterization of SOFC

After the durability tests for 400 hours at 850°C of a cell with $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_4$, a XRD analysis was made. The results are shown in Fig. 4.

The data from XRD analysis are summarized in Table 1.

The results in Table 1 show that there are derivative products, (i.e. $\text{CuSr}_2\text{O}_{3.43}$, $\text{CeSr}_7\text{Ni}_4\text{O}_{15}$ and SrNiO_3) as result of partial co-fusion of the CGO interlayer and the NdSrNiO_4 cathode, caused by the high temperature (850°C) of the durability tests. The SOFC can be considered as electrochemical devices, due to the typical features of the electrochemical sources of electricity that they possess. Due to this fact, on the cathodes of the SOFC reduction processes occur, in similarity to the classical Galvani elements. Because of the reduction on the SOFC cathode, this process is accompanied by partial reduction of the cathode composing material NdSrNiO_4 , leading to obtaining of metallic Nickel.

Thus, the comparison of the data, obtained by the

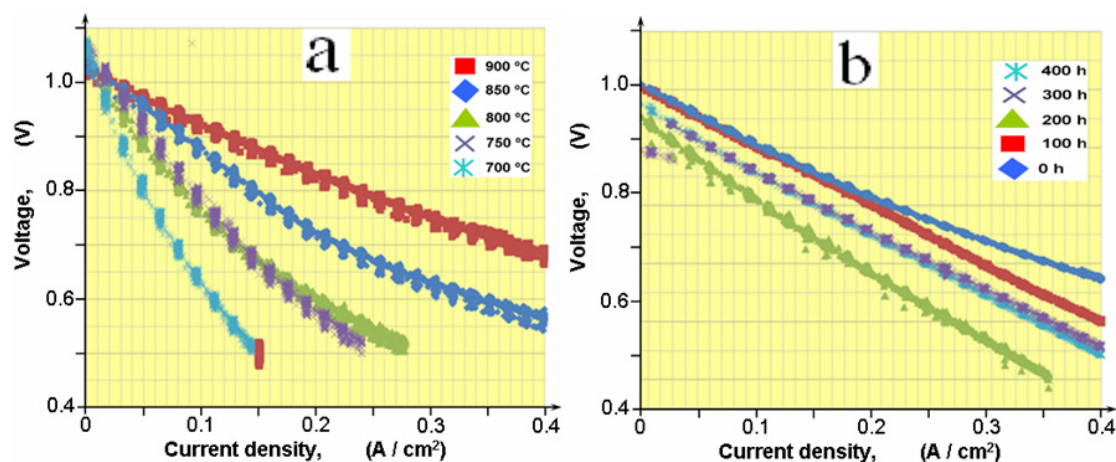


Fig. 3. Temperature (a) and time (b) dependences, recorded for assembled SOFC.

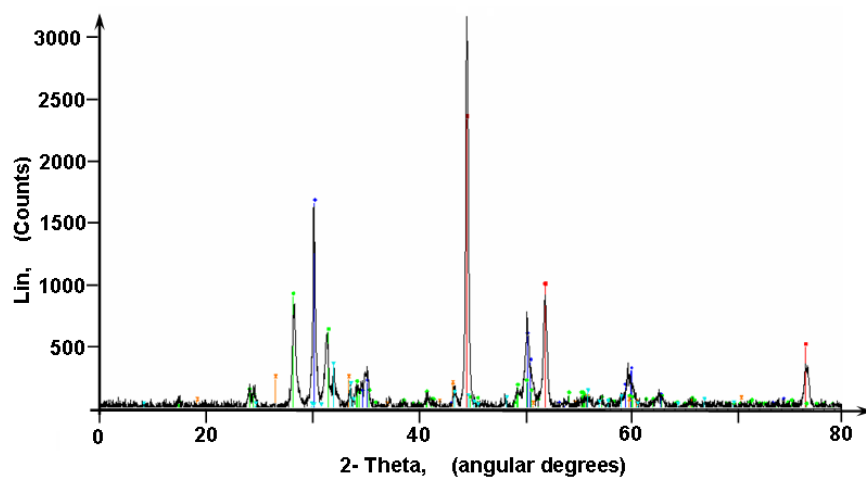


Fig. 4. XRD patterns of $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_4$ in the fuel cell after 400 hours of exposition at 850°C .

durability tests (Fig. 2(b)) and the results of the XRD analysis after these tests (Figs. 3, 4 and Table 1), leads to the conclusion that during the durability tests, the cathode material suffers a partial decomposition. It takes place simultaneously with high temperature chemical reactions with the components of the CGO interlayer. The process leads to coincident reduction of the Ni-compounds to metallic Ni, and subsequent oxidation to NiO, resulting in an increase of the cathodic R_p , as was discussed above. Nevertheless, after the initial 100 hours of the durability tests, the cathode material retains its parameters, indicating remarkable durability. This fact shows that the Ni stays in a permanent equilibrium between its reduced form (metallic Ni), and its oxidized form (NiO). This equilibrium is an evidence for constant simultaneous presence reduction and oxidation of this element – predetermining the maintenance of the

electrical properties of the cathodic material during the durability tests.

The SEM investigations also give evidence for the compatibility of the NdSrNiO_4 cathode material with the CGO interlayer. Fig. 5 shows a SEM cross-sectional image of a cell after 400 hours of durability tests.

There is not any kind of cracking between the NdSrNiO_4 cathodic material and the CGO interlayer, as can be seen from the figure. This fact confirms that there is a partial diffusion between these two materials, accompanied by formation of additional derivative phases, such as: $\text{CuSr}_2\text{O}_{3.43}$, $\text{CeSr}_7\text{Ni}_4\text{O}_{15}$ and SrNiO_3 . The absence of whatever cracks, ruptures and disjoining between the cathode and the interlayer reveals the compatibility between these materials. This fact is an indication for the lack of whatever mechanical tensions between these materials due to either their flexibilities or

Table 1. Crystalline phases determined by XRD after 400 hours of durability tests of $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_4$ cell.

Phase	Crystalline lattice	Parameters of the elementary cell, Å	Edges of the elementary cell, °
Ni	Cubic; wall centered Fm-3m (225)	a = 3.52; b = 3.52; c = 3.52	$\alpha = 90; \beta = 90; \gamma = 90$
$\text{Zr}_{0.92}\text{Y}_{0.08}\text{O}_{1.96}$	Elementary tetragonal – P42/n	a = 3.61; b = 3.61; c = 5.17	$\alpha = 90; \beta = 90; \gamma = 90$
ZrO_2 (badellite)	Elementary Monoclinic stable; – P21/a (14)	a = 5.31; b = 5.21; c = 5.15	$\alpha = 90; \beta = 99.22; \gamma = 90$
$\text{CuSr}_2\text{O}_{3.43}$	Tetragonal; bulk centered– 14/m	a= 3.77; b= 3.77; c= 13.25	$\alpha = 90; \beta = 90; \gamma = 90$
SrNiO_3	Hexagonal	a = 5.36; b = 5.36; c = 4.86	$\alpha=90; \beta=90; \gamma=120-120.7$
$\text{Zr}_{0.92}\text{Y}_{0.08}\text{O}_{1.96}$	Elementary Tetragonal – P42/n	a = 3.61; b = 3.61; c = 5.17	$\alpha = 90; \beta = 90; \gamma = 90$

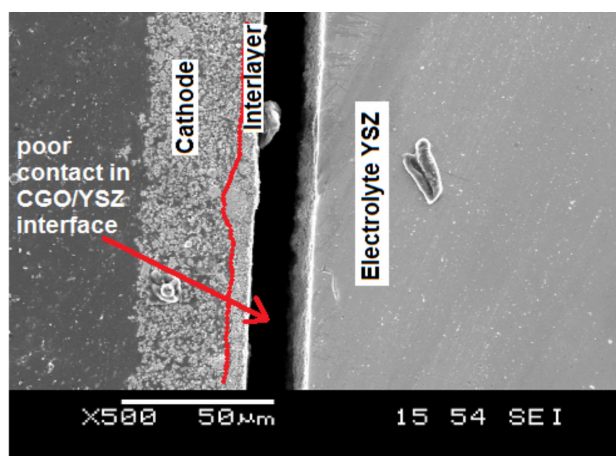


Fig. 5. SEM cross-sectional image of a cell after 400 hours of durability tests.

equal coefficients of thermal expansions (CTE).

However, there is a large crack between the CGO interlayer, and the YSZ solid electrolyte. This fact is an indication for the need to extend the thickness of the interlayer, in order to avoid the tensions that appear in the CGO / YSZ interface. The most probable reason for the appearance of these tensions is the significant difference between the CTE of both of NdSrNiO_4 and YSZ. The thicker GCO layer should provide efficient mechanical buffer to decrease the difference between the CTE of the cathode and the electrolyte.

CONCLUSIONS

After the analysis of the results of the present work, the following conclusions could be done:

Strontium doped neodymium nickelate ($\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-d}$) was tested as potential alternative cathode material for SOFC applications. The experiments included tests for determination of the electrical characteristics performed at 700 to 900°C and durability tests at 850°C and 0.5 A/cm², for up-to 400 hours.

After the respective tests, the cells were submitted to XRD and SEM characterizations.

The experiments conduct for determination of the electrical characteristics revealed that the $\text{Nd}_{1.6}\text{Sr}_{0.4}\text{NiO}_4$ is the optimal composition among the investigated compounds.

The optimal combination of initial voltage and current density were registered for the cell, submitted to 900°C.

The durability tests of the cell revealed negligible changes during the initial 100 hours of exposition to 850°C and charging by 0.5 A/cm² and afterwards it retains its characteristics, revealing a high durability. This fact is an evidence for the excellent performance of the $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-d}$ and its remarkable compatibility with CGO during the durability tests.

In order to improve the durability of the entire cell it is recommendable to increase the thickness of the CGO interlayer between the $\text{Nd}_{2-x}\text{Sr}_x\text{NiO}_{4-d}$ cathode and YSZ solid electrolyte.

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